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TEMPERATURE INDUCED DEGRADATION OF Nb Ti/Cu COMPOSITE SUPERCONDUCTORS

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The degradation mechanisms of state-of-the-art Nb-Ti/Cu superconductors are described, based on in-situ synchrotron X-ray diffraction measurements during heat treatment. A quantitative description of the Nb-Ti/Cu degradation in terms of critical current density, Cu stabiliser resistivity and mechanical composite strength is presented. In an applied magnetic field a significant critical current degradation is already observed after a 5-minute 400 °C heat treatment, due to variations of α -Ti precipitate size and distribution within the Nb-Ti alloy filaments. A strong degradation of the strand mechanical properties is observed after several minutes heating above 550 °C, which is also the temperature at which the formation of Cu Ti intermetallic phases is detected. Several minutes heating at 250 °C are sufficient to increase the RRR of the strongly cold work strands inside a Rutherford type cable from about 80 to about 240. Heating for several minutes at 400 °C does not cause a significant conductor degradation in self-field and, thus, leaves enough temperature margin for the electrical interconnection of Nb-Ti/Cu conductors with common low temperature solders.

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Temperature induced degradation of Nb-Ti/Cu composite superconductors

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Abstract. The degradation mechanisms of state-of-the-art Nb-Ti/Cu superconductors are described, based on *in-situ* synchrotron X-ray diffraction measurements during heat treatment. A quantitative description of the Nb-Ti/Cu degradation in terms of critical current density, Cu stabiliser resistivity and mechanical composite strength is presented. In an applied magnetic field a significant critical current degradation is already observed after a 5-minute 400 °C heat treatment, due to variations of α -Ti precipitate size and distribution within the Nb-Ti alloy filaments. A strong degradation of the strand mechanical properties is observed after several minutes heating above 550 °C, which is also the temperature at which the formation of Cu-Ti intermetallic phases is detected. Several minutes heating at 250 °C are sufficient to increase the RRR of the strongly cold work strands inside a Rutherford type cable from about 80 to about 240. Heating for several minutes at 400 °C does not cause a significant conductor degradation in self-field and, thus, leaves enough temperature margin for the electrical interconnection of Nb-Ti/Cu conductors with common low temperature solders.

1. Introduction

Nb-Ti/Cu composite superconductors have so far been used for the construction of all superconducting particle accelerators, mainly for the excellent mechanical properties of the ductile composite strands made of typically several thousand Nb-Ti alloy filaments inside a high purity copper matrix. As an example, for the fabrication of the Large Hadron Collider (LHC) main magnet coils 220 000 km of Nb-Ti/Cu strand have been manufactured [1]. The extracted Nb-Ti alloy filaments have an ultimate tensile strength of about 1.2 GPa [2].

During a series of cold work and heat treatment (HT) steps the α -Ti precipitate size and distribution within the Nb-Ti alloy filaments is optimised for maximum critical current density (J_c) at the operating magnetic field [3]. The finished Nb-Ti/Cu strands and cables may then be subjected to additional HT, for instance for the annealing of the cold-worked Cu matrix prior to coil winding, or for electrical interconnection by soldering. Therefore, it is of interest how these HT's modify the composite strand superconducting, normalconducting and mechanical properties. The influence of additional HT's on the current carrying capability of Nb-Ti superconductors has been described for instance in [4]. It has been shown that 2-minute 400 °C post annealing of a Nb-Ti strand can significantly reduce the critical

current in an applied magnetic field. The main goal of this work is to describe the temperature induced modifications of the strand properties in self field.

The strand studied, which is used for the fabrication of the LHC main dipole magnets outer layer (so-called 02 type), is optimized for achieving best J_c at the LHC nominal field of 8.3 Tesla. The strand has a nominal diameter of 0.825 mm, a Cu/SC ratio of 1.95 and a twist pitch length of 15 mm. The reference strand used is uncoated. In order to study the variations of the Cu stabiliser resistivity inside Rutherford type cables, Sn-Ag coated strand samples were extracted from a LHC 02 cable.

The strand samples were characterised by synchrotron X-ray diffraction measurements during *in-situ* HT, magnetisation measurements, Residual Resistivity Ratio (RRR) measurements and tensile tests. For all HT's the temperature ramp rate was 200 °C/h, and the dwell for the *ex-situ* HT's was always 5 minutes.

2. Results

2.1. Ti diffusion into the Cu stabiliser and formation of Cu-Ti intermetallic phases

The formation of new phases during the Nb-Ti/Cu strand HT has been monitored by synchrotron X-ray diffraction at the high energy scattering beam line ID 15 of the European Synchrotron Radiation Facility (ESRF). The high flux of high energy X-rays provided through ID15 allows to record diffraction patterns with excellent signal-to-noise-ratio, even for small amounts of weakly diffracting phases. Further details about the diffraction experiment can be found in [5].

During an HT in a dedicated X-ray transparent furnace, Debye Scherrer diffraction patterns of the Nb-Ti/Cu strand were continuously acquired in transmission geometry with an X-ray energy of 86.8 keV. The colour intensity plot presented in figure 1 shows a sequence of 55 radially integrated diffractograms that have been acquired during the *in-situ* HT with a ramp rate of 200 °C/h.

It can be seen that Cu_4Ti_3 and Cu_4Ti are first detected at a temperature of about 550 °C. Additional intermetallic phases (believed to be Cu_2Ti and/or Cu_3Ti_2) are detected at about 700 °C. Claim of the existence of these two phases is tentative because their strongest peaks overlap with prominent peaks of other phases such as Cu_4Ti , Cu_4Ti_3 and Cu. Metallographic examination of heat treated strand samples shows that Ti diffuses into the Cu matrix, and also that Cu diffuses into the Nb-Ti filaments. At about 900 °C low melting Cu-Ti compounds decompose into liquid phases [6]. The fine structure of the intermetallic phases, which is sub-micron, prevented the exact composition from being investigated by SEM-EDS.

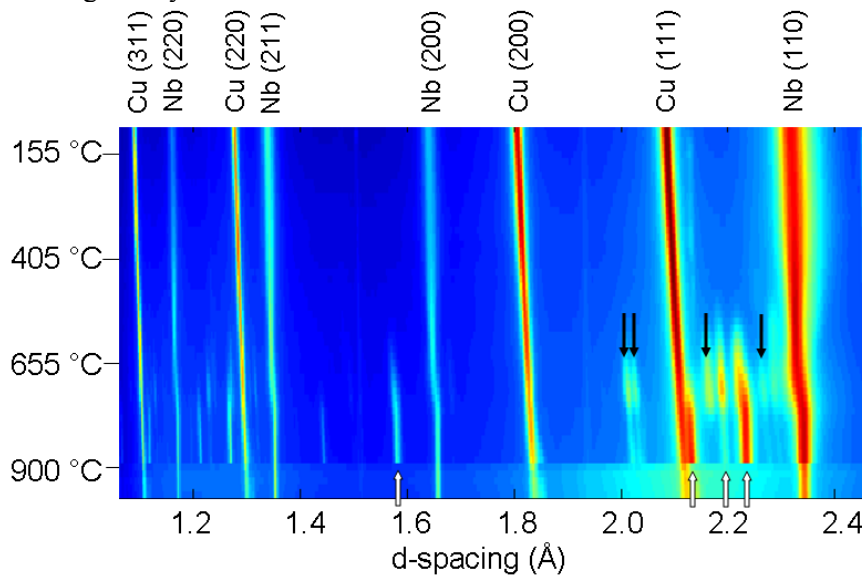


Figure 1: Sequence of 55 radially integrated diffractograms acquired during HT of the Nb-Ti/Cu strand with a ramp rate of 200 °C/h. Cu_4Ti_3 and Cu_4Ti peaks are labeled with open and closed arrows, respectively.

2.2. Temperature induced degradation of the critical current density

In order to investigate the high temperature annealing effect on the critical current density (J_c), magnetization measurements at $T = 4.2$ K from -6 to +6 T were performed using a Vibrating Sample Magnetometer (VSM).

In the presence of flux pinning the sign of the superconducting magnetization depends on the direction of the field sweep, thus resulting in a hysteresis loop between the ascending and the descending branches of the magnetization. The irreversible magnetization ΔM is defined as $(M^+ - M^-) / 2$, where M^+ (M^-) is the branch of the magnetization for $dB_a/dt < 0$ ($dB_a/dt > 0$). B_a is the applied field, swept at a constant rate: in our case $|dB_a/dt| = 2$ T/min. ΔM is proportional to J_c times a geometrical factor.

As shown in figure 2, magnetization measurement revealed noticeable degradation of ΔM , and thus of J_c , in an applied field after post annealing. For the heat treatment at 400 °C, at 3 T ΔM is already reduced by more than 20%. This indicates a loss of flux pinning.

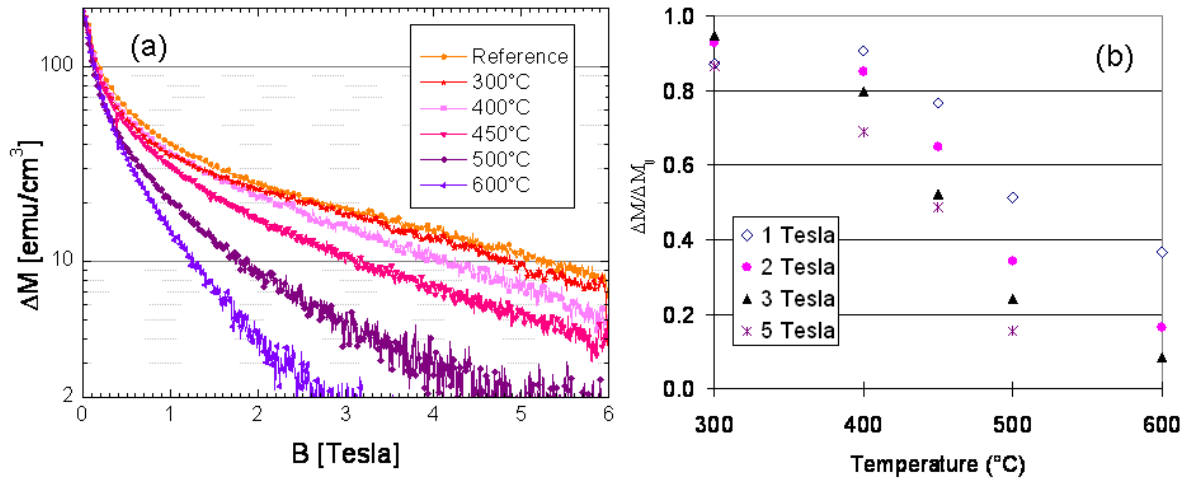


Figure 2: Variation of the Nb-Ti/Cu strand magnetization (ΔM) at 4.2 K in magnetic field up to 6 T after different HT's (a) and relative flux pinning reduction vs. annealing temperature at different magnetic field (b).

2.3. Degradation of the composite tensile properties

The temperature induced variations of the mechanical properties have been examined by measuring the ultimate tensile strength (R_m), the elongation at fracture (ε) and the E-modulus (E_a) of the composite strand in longitudinal direction (Young's modulus) by uniaxial tensile tests. E_a is determined from the unloading stress strain curves. Initially ε increases with temperature, due to an annealing of the Cu matrix. As shown in figure 3(a), ε decreases during the 550 °C HT, due to Ti diffusion into the Cu matrix and formation of brittle Cu-Ti intermetallic phases. When heated above 600 °C the composite strand exhibits brittle behaviour. The ultimate tensile strength of the as-received composite is 690 MPa. A slight R_m decrease is observed at relatively low temperature, due to Cu annealing. HT's above 600 °C strongly reduce R_m .

The increase of E_a by about 10 % with temperature (see figure 3 (b)) is at least partly attributed to the increased amount of α -Ti [2], which is precipitated during the HT (after identical degree of cold work the α -Ti content increases with temperature and HT duration [3]). A change of the preferential Cu grain orientation from a $\langle 111 \rangle$ fibre texture, which is developed during cold work, to a $\langle 111 \rangle$ and $\langle 200 \rangle$ duplex texture during the HT [7] may also vary the strand E-modulus.

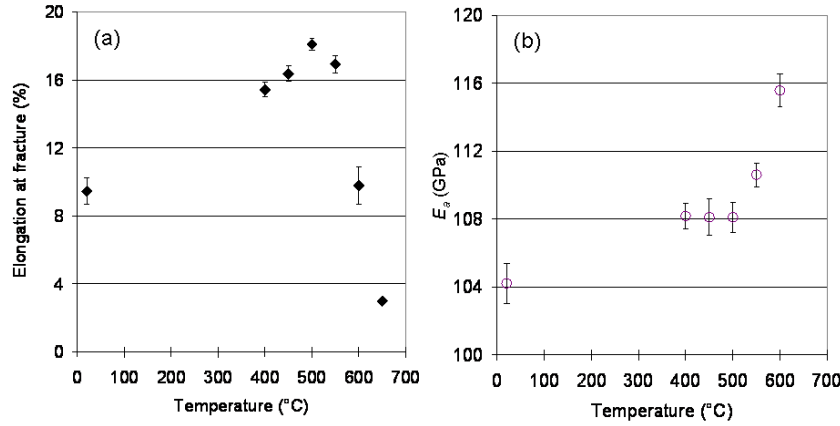


Figure 3: Composite strand elongation at fracture vs. peak temperature (a) and E-modulus vs. peak temperature (b). Results are the average values of 3 measurements $\pm 1\sigma$.

2.4. Variation of the composite electrical conductivity

The HT influence on the electrical resistivity of the Cu stabiliser has been assessed by RRR measurements (defined as the ratio of the strand resistance measured at 293 K to that at 10 K). As shown in figure 4, the RRR of the uncoated, non-cabled strand initially increases with temperature due to Cu annealing. When the temperature exceeds 550 °C the RRR decreases with increasing temperature, possibly because of Ti diffusion into the stabiliser. A similar RRR vs. temperature behaviour as observed here for the Nb-Ti/Cu composite, has also been reported for pre-annealed oxygen-free copper [8].

RRR measurements of strand extracted from LHC cables before final cable annealing show that during the cabling process the RRR of the strand type studied here is reduced from typically 170 to about 80 [9].

Due to the different degree of cold work along the strands extracted from keystoneed Rutherford type cables, the RRR result obtained for the 80 mm-long sample is an average over the probed strand length. The RRR reduction during cabling is particularly strong at the thin cable edge where the Cu stabiliser undergoes severe cold work. In figure 4 it can be seen that the effect of the post annealing of strand extracted from a 02 cable is initially stronger than it is for the non-cabled strand. After 5 min-250 °C HT the RRR of the extracted strand is increased from 80 to about 240. Sn diffusion from the strand coating into the Cu stabiliser may contribute to the degradation of the RRR during the 500 °C HT of the extracted strand.

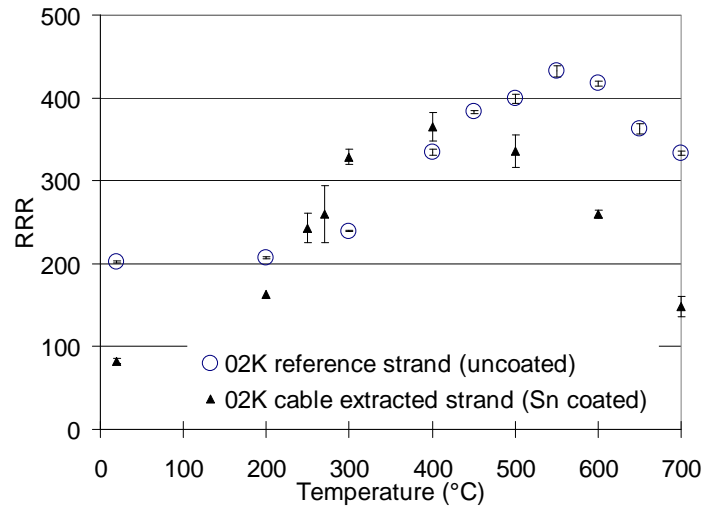


Figure 4: RRR of the reference strand and the strand extracted from a Rutherford cable vs. peak temperature. Results are the average values of 3 measurements $\pm 1\sigma$.

3. Discussion and conclusion

The degradation of Nb-Ti/Cu composite strands occurs in two steps. Already after 5 minutes heating at 400 °C J_c in an external field is significantly reduced. This J_c reduction is caused by changes of α -Ti precipitate size and spacing inside the Nb-Ti alloy filaments and hence loss of flux pinning force.

At temperatures exceeding 550 °C Ti diffusion through the Nb barriers and formation of CuTi intermetallics is observed, which degrades the composite mechanical properties. When heated for some minutes above 600 °C the composite shows brittle behaviour and loses entirely its load carrying ability.

The electrical interconnections of the LHC superconductors are made outside the magnet coils. Therefore, for an assessment of the superconductor modifications induced during electrical interconnection the changes of the conductor properties in self field are relevant.

Heating the strand for several minutes at 400 °C, which is well above the fusion temperature of common low temperature solders, causes only a slight J_c degradation in self field, improves the Cu stabiliser conductivity and does not degrade the ductile behaviour of the composite. Thus, there is substantial temperature margin for the production of electrical interconnections by low temperature soldering, provided the interconnections are not used in applied magnetic fields. The maximum temperature limit for Nb-Ti strands that are operating in an applied field is much lower than that for those operating in self field.

Acknowledgements

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